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GOVERNMENT OF INDIA

MINISTRY OF FOOD AND AGRICULTURE

Directorate of Marketing and Inspection

METHODS OF SAMPLING AND TESTING VEGETABLE OILS AND FATS UNDER AGMARK

DECEMBER 1952

Issued by the Agricultural Marketing Adviser to the Government of India, New Delhi

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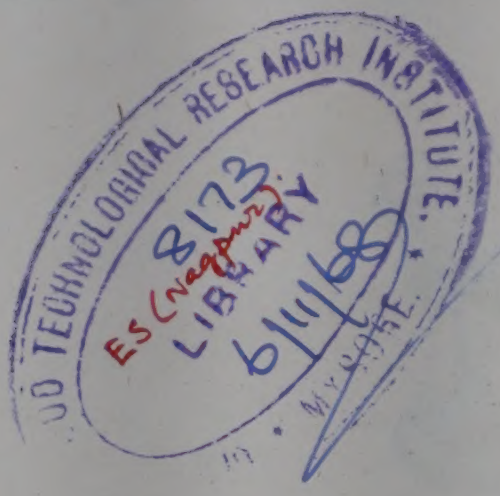

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SCOPE.

The methods of sampling and testing embodied in this publication are intended principally for the use of packers authorised to grade vegetable oils and fats on the basis of Agmark standards of quality prescribed under the Agricultural Produce (Grading and Marking) Act, 1937.

(This cancels all previous issues on the subject)

CONTENTS.

	Pages
Part A—Sampling.	
1. General principles	1
2. Apparatus required	1-2
3. Number of samples	2
4. Size of samples	2
5. Procedure	2 3
6. Marking and despatch of sample containers ..	4
7. Note	4
Part B—Analysis.	
I.—*General tests.*	
1. Determination of colour	4
2. Determination of specific gravity	4 7
3. Determination of refractive index	7-9
4. Determination of acid value	9-10
5. Determination of saponification value	10-11
6. Determination of iodine value	11-14
7. Determination of unsaponifiable matter	15-16
8. Methods for detecting the presence of different oils	16
(A) Detection of mineral oil	16-17
(B) Detection of cottonseed oil	17
(C) Detection of sesame oil	17-18
(D) Detection of groundnut oil	18-19
(E) Detection of argemone oil	19
(F) Detection of linseed oil	20-21

PAGES

II.—*Special tests.*

1. Mustard oil 21

(A) Determination of allyl iso-thiocyanate .. 21-22

(B) Detection and determination of hydrocyanic acid 22-24

2. Castor oil 24

(A) Determination of acetyl value 24-25

(B) Determination of critical solution temperature in alcohol 25-26

3. Vanaspati 26

Determination of melting point (capillary slip method) 26-27

Plates .. 4

PART A—SAMPLING.

1.—General principles.

(*a*) No amount of correct analysis can make up for incorrect sampling. It is, at the same time, almost impossible to write directions for sampling of oils and fats that will cover all conditions and circumstances that may confront the individual responsible for taking the sample. There are many instances in which the judgment and experience of the individual shall prevail. There are, however, certain general rules which must always be followed if the sample is to be representative.

(*b*) If the bottom of the tank from which sample is to be taken is smaller than its middle or top or *vice versa,* the contour of the tank should be taken into account. The number of samples from each section (*e.g.* each 1-foot level) should be regulated in reverse order to the cubical capacity of each section. For example, if the bottom 1-foot section is one-fourth of the middle 1-foot section, then one 1-foot sectional sample should be drawn from the bottom level and four 1-foot sectional samples from the middle. These samples are then thoroughly mixed and made into a composite sample.

(*c*) If the consignment is not uniform in character, each sample drawn should be treated as separate and submitted to the laboratory without mixing.

2.—Apparatus required.

(*a*) *Weighted sample bottle.*

A suitable type of weighted sample bottle is shown in fig. 1. It is normally of 2-3 lbs. capacity. The total weight of the bottle together with its carrier is such that it readily sinks into the oil. It is provided with means to permit filling at any desired level and is attached with a long cord or chain.

(*b*) *Sampling thief.*

A satisfactory type of thief is shown in fig. 2. It consists of a metal or thick walled glass tube which varies from $\frac{3}{4}''$ to $1\frac{1}{2}''$ in diameter and 15″ to 40″ in length. The upper and lower ends are conical and narrowed down to about $\frac{1}{4}''$ to $\frac{1}{2}''$. The rings attached to the upper end of the tube are to facilitate handling. The bottom end is tapered at such an angle that there is approximately $\frac{1}{4}''$ vertical distance between the top and the bottom of the taper *i.e.* 45° taper for $\frac{1}{4}''$ opening.

(*c*) *Trier for solid fats.*

It is a half-round metal tube $\frac{1}{2}''$ to 1″ in diameter and 2′ to 5′ in length depending upon the size of the package to be sampled. One end is tapered to a point, the taper to be not more than 1″ long. The other end is attached to a T-shaped handle. Copper, brass or bronze must not be used in the construction of this trier.

(*d*) *Sample containers.*

Metallic sample containers of 20 oz. (1¼ lbs.) capacity with suitable tight stoppers.

3.—NUMBER OF SAMPLES.

(*a*) *From tanks.*

Each tank is to be sampled separately and three samples are to be drawn from each tank as follows :—

(*i*) A top sample taken at a level 1|10th of the depth of the oil measured from the top.

(*ii*) A middle sample taken at a level one-half of the depth of the oil in the tank.

(*iii*) A bottom sample taken at a level 9|10th of the depth of the oil measured from the top.

The samples thus collected are to be thoroughly mixed together into one bulk sample.

(*b*) *From drums, barrels, tins and other packages.*

The minimum number of samples to be drawn is equal to the cube root of the number of packages (rounded to the next higher integer). For example, if there are 10, 50 or 125 drums in a consignment, the number of samples to be drawn is 3, 4 or 5 respectively. When, therefore, samples are to be drawn from packs, a number of packs equal to the number of samples to be drawn are to be selected at random and one sample drawn from each one of them. The samples are to be mixed together, thoroughly, into one sample.

4.—SIZE OF SAMPLES.

(*a*) In case of oils, the minimum quantity of each laboratory sample is 20 oz. (1¼ lbs.).

(*b*) In case of solid fats, the minimum quantity of each laboratory sample is 1 lb.

5.—PROCEDURE.

(*A*) (*a*) *Sampling from tanks—liquid contents.*

(*i*) Lower the stoppered weighted sample bottle 2 (*a*) to the appropriate depth in the tank, as required under 3(*a*) above.

(*ii*) Uncork the bottle by a quick jerk of the cord allowing it to fill completely at the appropriate level.

(*iii*) Withdraw the bottle immediately.

(*iv*) Collect each sample in a separate container.

(*v*) Mix the samples thoroughly and prepare a composite sample.

WEIGHTED SAMPLE BOTTLE

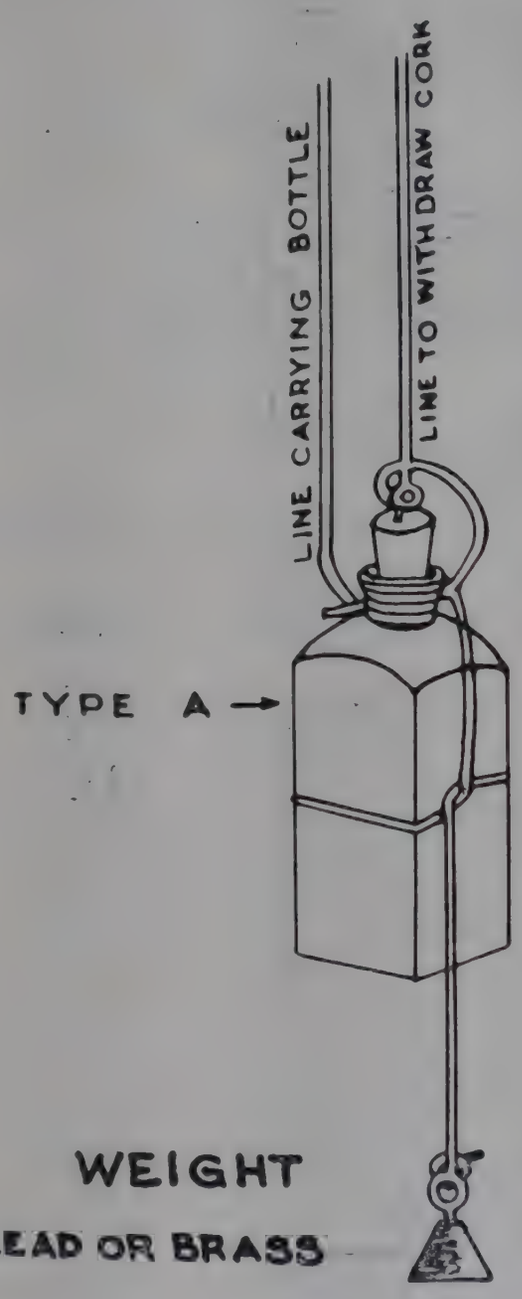

FIG. 1

6.—Marking and despatch of sample containers.

(*a*) As gummed labels on glass or metallic containers are likely to fall off, wherever possible, marking is to be done by stencilling. Alternatively, the label is to be tied to the container securely, with or without a duplicate gummed label. The following particulars on the sample are to be given :—

(*i*) Name and address of the person sending the sample.

(*ii*) Name and grade of oil.

(*iii*) Sample reference number.

(*iv*) Reference number of any letter or forwarding note giving more details.

(*b*) One sample properly marked and packed is to be sent to the laboratory for testing while the duplicate is to be retained by the sender till the report of the laboratory is received.

7.—Note.

(*a*) The individual responsible for collection of samples should safeguard against the tendency of getting the samples drawn by untrained or semi-trained subordinates.

(*b*) Metallic sample containers when used, and all sampling apparatus should be properly cleaned and dried before use.

(*c*) Glass containers for samples may be used when distant despatch is not involved, *i.e.*, in case of samples drawn at a place where a laboratory is available.

(*d*) For sending samples to long distances, the sample containers should preferably be packed in a wooden box and the space around the sample containers filled with saw dust or other absorbent material. Care should be taken that the nails do not pierce the containers.

PART B—ANALYSIS.

I—General tests.

1.—Determination of colour.

Colour should be determined by a Lovibond tintometer meant for oils and fats. The instructions supplied with the instrument should be strictly adhered to.

2.—Determination of specific gravity.

(*1*) *Definition.*

The specific gravity of a substance is the weight of any volume of the substance compared with the weight of an equal volume of pure water, at a standard temperature. Under Agmark this temperature is specified to be 30°C.

(2) *General remarks.*

There are many methods for determining the specific gravity of oils, out of which two methods, namely, the hydrometer method and the specific gravity bottle method have been adopted.

The hydrometer method should be followed as a routine method for rapid determination of specific gravity. Specific gravity which falls near about the limits prescribed under the specifications should be determined by the specific gravity bottle method.

(A) HYDROMETER METHOD.

(1) *Outline.*

The hydrometer is floated in a sufficient quantity of the oil contained in a glass cylinder and the specific gravity read off directly on the scale placed inside the stem.

(2) *Apparatus required.*

(*a*) A set of three standard specific gravity hydrometers in the 50 degree series reading from 0.850 to 0.900, 0.900 to 0.950 and 0.950 to 1.000 respectively with 0.001 as the smallest division of the scale. Use of a set standardised at a temperature as near as possible to 30°C is preferred.

(*b*) An ungraduated glass cylinder having an internal diameter one inch more than the external diameter of the hydrometer at its widest point.

(*c*) A certified thermometer of any convenient range graduated to $\frac{1}{2}$°C.

(3) *Procedure.*

(*a*) Clean and dry the glass cylinder and fill to the necessary depth with the oil sample, avoiding air bubbles as far as possible.

(*b*) Clean and dry the hydrometer and immerse carefully in the oil so that it floats freely in it without touching either the sides or the bottom of the cylinder.

(*c*) When the hydrometer is steady, depress it momentarily into the oil by about $\frac{1}{2}$ cm. and release.

(*d*) When the hydrometer is steady again, read the scale at the lower meniscus of the oil. In order to avoid parallax error, place the eye in the same level with the surface of the liquid while taking the reading.

(*e*) Ascertain the temperature of the oil and make a correction so as to obtain the result at 30°C.

(4) *Calculation of result.*

The corrected specific gravity (S) at 30°C|30°C should be obtained by the following formula :—

$$S = St \times \frac{D_1}{D_2} + 0.00064 \ (t - 30).$$

where St is the observed reading at temperature t and D_1 and D_2 are the densities of water at the temperature at which the hydrometer is standardised and at 30°C, respectively.

(*5*) *Note.*

(*a*) The hydrometer should be checked either by comparison with a certified hydrometer or with the help of a liquid whose specific gravity has been determined by the specific gravity bottle method.

(*b*) The hydrometer and cylinder should be cleaned with warm soft soap solution and wiped dry with a soft cloth when the work is finished.

(B) Specific gravity bottle method.

(*1*) *Outline.*

The specific gravity bottle is first weighed empty and then after filling with water. It is next weighed when filled with the oil. The net weight of oil is then divided by that of water to give the specific gravity.

(*2*) *Apparatus required.*

(*a*) A 50 ml. capacity specific gravity bottle along with thermometer, as shown in fig. 3.

(*b*) A piece of clean muslin.

(*3*) *Procedure.*

(*a*) Clean and dry the bottle along with its thermometer and stopper.

(*b*) Insert the thermometer and stopper and weigh the bottle accurately. Note weight (P) gms.

(*c*) Fill the bottle with distilled water taking care that no air bubbles are introduced into it. Insert the thermometer.

(*d*) Allow the bottle to stand in a bath maintained at 30°C ±0.5°C for at least half an hour.

(*e*) Adjust the level of water in the bottle upto the mark and stopper the bottle, wiping any superfluous water which may be sticking to the sides of the neck.

(*f*) Remove the bottle from the bath and wipe dry with a muslin piece. Weigh the bottle and note weight (Q) gms.

(*g*) Repeat the process from (*a*) to (*f*) by using the oil to be tested *i.e.* clean and dry the bottle, fill with the oil, maintain at 30°C ±0.5°C for ½ an hour, adjust the level, wipe dry, weigh and note the weight (R) gms.

(*h*) Empty oil back into the oil bottle. Clean the bottle with hot soft soap solution and dry.

(*4*) *Calculation of result.*

Weight of the oil at 30°C = R—P

Weight of water at 30°C = Q—P

Specific gravity of the oil at

$$30°C|30°C = \frac{R—P}{Q—P}$$

SPECIFIC GRAVITY BOTTLE WITH THERMOMETER

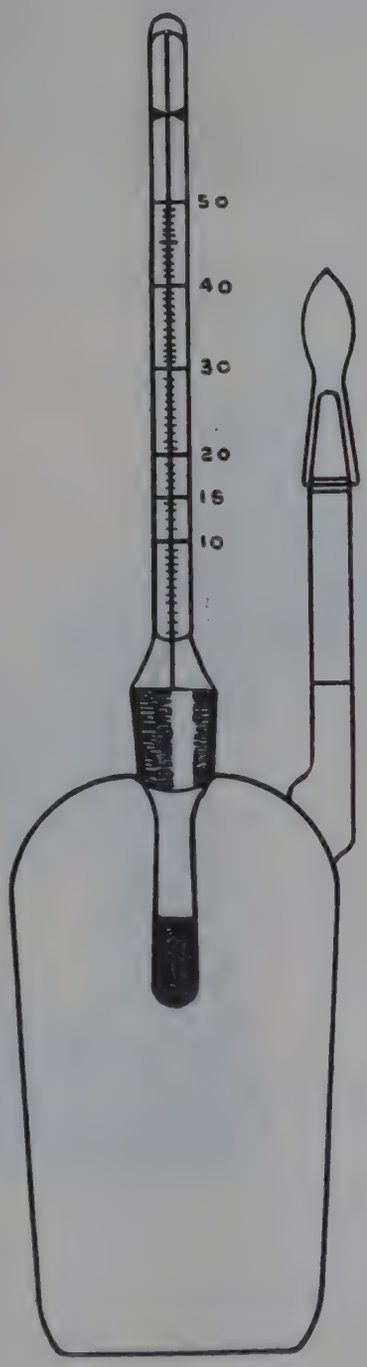

FIG. 3

THE ABBE REFRACTOMETER

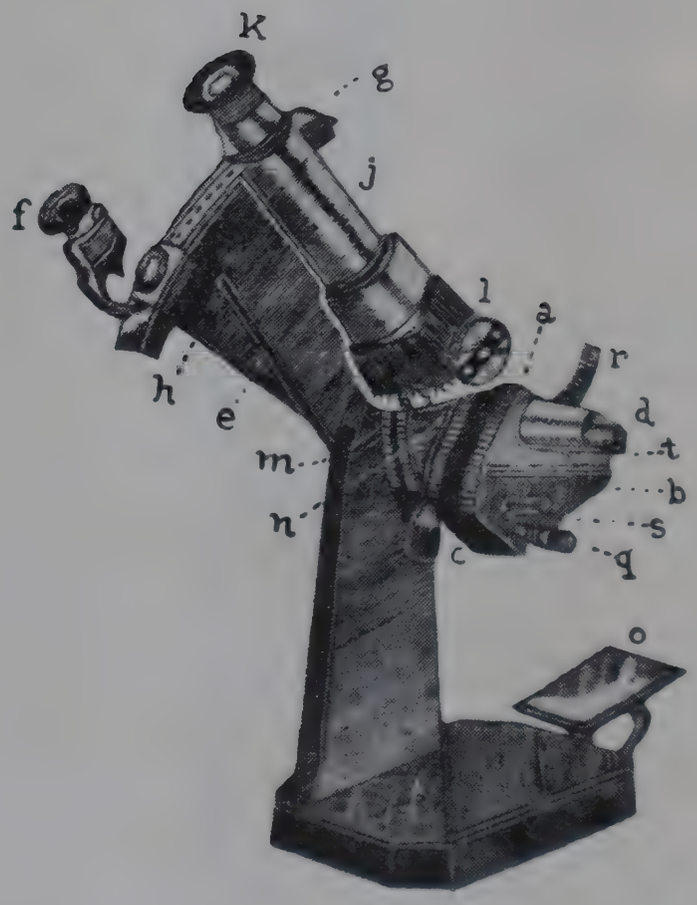

FIG. 4

(5) *Note.*

(*a*) The weight of water (Q—P) serves as a constant for the bottle and is used in all determinations of specific gravity. This constant, however, should be checked periodically—say once in two months.

(*b*) The specific gravity of the oil may be determined at the water temperature (t) provided this temperature is within ± 5°C of 30°C and the corrected specific gravity (S) at 30°C|30°C obtained by the following formula :—

$$S = St + (t-30) \times 0.00064.$$

when St is the observed specific gravity at temperature t. For very accurate result, however, oil must be weighed at 30°C.

(*c*) The drying of the bottle is rapidly performed by rinsing with small quantities of dry alcohol followed by small quantities of dry ether, draining the latter from the bottle and blowing dry air into it.

3.—Determination of refractive index.

(1) *Definition.*

The refractive index is a number expressing the ratio between the velocity of light in air and its velocity in some other medium.

(2) *General remarks.*

Any form of refractometer may be used provided it is capable of giving results to an accuracy of 0.0002 and the temperature of the sample can be controlled within ± 0.5°C. Whichever instrument is used, its accuracy should be periodically checked with a liquid of known refractive index. For determining the refractive indices of oils, Abbe refractometer is a common and convenient instrument. For accurate determination which is necessary in the case of marginal (readings) values, the refractive index should be determined at the standard temperature, namely, 40°C.

(3) *Description.*

A brief description of the Abbe refractometer is given below :—

Fig. 4 shows the instrument : **a** and **b** are prisms of dense flint glass mounted in water jackets, and can be clamped together by turning the milled head **c**. The face of the upper prism in contact with the liquid is polished, while the corresponding surface of the lower prism is left grey to prevent the formation of false images. A thermometer screws into position close to the nozzle at **d** and records the temperature of the circulating water. The prism system can be rotated about a horizontal axis by means of the arm **e**, to which is attached the reading lens **f** and the index, which moves over the scale **g**. The telescope **j** is rigidly attached to the sector **h** and this together with the prisms can be turned about a horizontal axis, the extreme positions being determined by stops. Light is reflected into the prisms by means of the mirror **o**. A compensator is attached to the end of the telescope, the prisms being rotated by the milled head **l** and their position recorded by the scale **m**. The lower prism can be rotated relatively to the scale by the black milled ring **n**

(*4*) *Preparatory arrangements.*

In order to obtain correct readings, the instrument is checked by means of the glass, test-piece supplied, having the refractive index engraved upon it. The reading is made as follows :—The sector **h** is moved over against the forward stop, the prism jackets opened, and the instrument turned round so that the telescope points towards the light source. The test-piece is now placed with its larger polished face on the prism face, a drop of liquid of higher refractive index (mono-bromo-naphthalene is recommended) being used to make the optical contact. The dividing line of the field is now achromatised by means of the compensator. The line is then set on the inter-section of the cross-webs by moving the index arm, and reading noted. If the latter does not agree with the value engraved on the test-piece, the prism jackets must be moved relative to the prism arm, three screws being gently loosened for this purpose and retightened when the setting is correct.

The instrument is connected with a thermostat so that a stream of water passes around the prisms at the required temperature. The water should enter at the nozzle **q** and leave at **r**, the other two nozzles, **s** and **t**, being connected by rubber tubing.

(*5*) *Procedure.*

(*a*) See that the thermometer has remained steady at the required temperature for 5-10 minutes.

(*b*) Push sector **h** over to front stop, opening prism jackets and moving index arm **e** if necessary until the face of the prism is horizontal

(*c*) Wipe the surface with a clean soft cloth, place a drop or two of oil on, close the jacket over and fix by turning milled head **c**. Restore sector to normal position, and direct a beam of light through the telescope by adjustment of the mirror.

(*d*) Focus the cross-webs sharply by means of eye-piece **k**. On turning the index arm **e**, the dividing line of the field will be indicated by a patch of bright colour between the light and dark parts of the field. Remove this by turning the milled head **l**, when the dark half of the field will appear bounded by a sharp edge accompanied by a purplish border.

(*e*) By a further movement of the index arm bring the sharp edge directly under the intersection of the cross-webs.

(*f*) Read the refractive index directly on the scale, using the magnifier **f** for this purpose. The third decimal place is read directly on the divided arc and the fourth decimal may be estimated accurately within two units.

(*6*) *Temperature correction.*

In case the thermostat is not used the refractive index is determined at the room temperature. The refractive index reading taken can be corrected to refractive index at 40°C. The average correction for temperature for oils is 0.00038 for each degree centigrade, so that 0.00038 is added or subtracted for each degree by which the temperature of observation is above or below 40°C.

(7) *Note.*

Refractive index can also be determined by the Butyro-refractometer provided the refractive index of the oil falls within the range of the instrument. It will be advisable in this case to use monochromatic sodium light for facilitating observation. The scale reading on the Butyro-refractometer should be converted into refractive index by reference to the following table :—

Table for conversion of scale readings (butyro-refractometer) into refractive indices nD.

Scale Reading.	Refractive index nD.	Scale Reading.	Refractive index nD.	Scale Reading.	Refractive index nD.	Scale Reading.	Refractive index nD.	Scale Reading.	Refractive index nD.
30·0	1·4452	40·0	1·4524	50·0	1·4593	60·0	1·4659	70·0	1·4723
30·5	1·4456	40·5	1·4528	50·5	1·4597	60·5	1·4663	70·5	1·4726
31·0	1·4460	41·0	1·4531	51·0	1·4600	61·0	1·4666	71·0	1·4729
31·5	1·4463	41·5	1·4535	51·5	1·4604	61·5	1·4669	71·5	1·4732
32·0	1·4467	42·0	1·4538	52·0	1·4607	62·0	1·4672	72·0	1·4736
32·5	1·4470	42·5	1·4542	52·5	1·4610	62·5	1·4675	72·5	1·4739
33·0	1·4474	43·0	1·4545	53·0	1·4613	63·0	1·4679	73·0	1·4742
33·5	1·4477	43·5	1·4549	53·5	1·4616	63·5	1·4682	73·5	1·4745
34·0	1·4481	44·0	1·4552	54·0	1·4620	64·0	1·4685	74·0	1·4748
34·5	1·4485	44·5	1·4556	54·5	1·4623	64·5	1·4688	74·5	1·4751
35·0	1·4488	45·0	1·4559	55·0	1·4626	65·0	1·4691	75·0	1·4754
35·5	1·4492	45·5	1·4563	55·5	1·4629	65·5	1·4694	75·5	1·4757
36·0	1·4495	46·0	1·4566	56·0	1·4633	66·0	1·4698	76·0	1·4760
36·5	1·4499	46·5	1·4569	56·5	1·4637	66·5	1·4701	76·5	1·4763
37·0	1·4502	47·0	1·4573	57·0	1·4640	67·0	1·4704	77·0	1·4766
37·5	1·4506	47·5	1·4577	57·5	1·4643	67·5	1·4707	77·5	1·4769
38·0	1·4510	48·0	1·4580	58·0	1·4646	68·0	1·4710	78·0	1·4772
38·5	1·4514	48·5	1·4584	58·5	1·4649	68·5	1·4713	78·5	1·4775
39·0	1·4517	49·0	1·4587	59·0	1·4653	69·0	1·4717	79 0	1·4778
39·5	1·4521	49·5	1·4590	59·5	1·4656	69·5	1·4720	79·5	1·4781

4.—DETERMINATION OF ACID VALUE.

(1) *Definition.*

The acid value of an oil or fat is the number of milligrammes of potassium hydroxide required to neutralize the free fatty acids present in one gramme of the oil or fat.

(*2*) *Outline.*

A known weight of oil is warmed with neutral alcohol and titrated with standard caustic soda solution using phenolphthalein as indicator.

(*3*) *Apparatus and chemicals required.*

(*a*) Accurate burettes and pipettes.

(*b*) Erlenmeyer flasks, 250 ml. capacity.

(*c*) N|10 sodium hydroxide solution.

(*d*) Accurately neutralised alcohol.

(*e*) 1% phenolphthalein solution in neutral alcohol.

(*4*) *Procedure.*

(*a*) Weigh accurately about 10 gms. of the sample in a 250 ml. Erlenmeyer flask, taking care that no oil adheres to the neck of the flask. Note weight (W) gms.

(*b*) Add 50 ml. of neutral alcohol and digest in a water-bath for about 15 minutes, agitating the flask now and then.

(*c*) Allow the flask to cool.

(*d*) Add 0.5 ml. of phenolphthalein solution and titrate with N|10 sodium hydroxide. Add the alkali drop by drop agitating the flask during addition until the pink colour persists for one minute.

(*e*) Note the number of ml. of alkali consumed (X) ml.

(*5*) *Calculation of result.*

$$\text{Acid value} = \frac{X \times 5.61}{W}$$

5.—Determination of saponification value.

(*1*) *Definition.*

The saponification value of an oil or fat is the number of milligrammes of potassium hydroxide required to completely saponify one gramme of the oil or fat.

(*2*) *Outline.*

A known weight of the sample is refluxed with excess alcoholic potash and the unabsorbed potash is determined by back titration with N|2 hydrochloric acid ; a blank determination is also carried out and the saponification value is calculated from the difference between these two readings.

(*3*) *Apparatus and chemicals required.*

(*a*) Two Erlenmeyer flasks, 250 ml. capacity (both flasks should be of the same glass preferably Jena or Pyrex).

(*b*) Two upright reflux condensers on stands.

(*c*) *Purified alcohol.*—Dissolve 1.5 gms. of silver nitrate in about 3 ml. of distilled water, add this to 1 litre of 95% ethyl alcohol contained in a glass stoppered bottle and mix thoroughly. Dissolve 3 gms. of potassium hydroxide in 10-15 ml. of warm alcohol, cool this solution, add

it slowly to the alcoholic silver nitrate, stirring slightly. Allow the precipitated silver oxide to settle, siphon off the clear supernatant solution and distil the alcohol on a steam bath.

(*d*) *Alcoholic potash solution.*—Approx. N|2. Dissolve 30 gms. of potassium hydroxide in 1 litre of the purified alcohol and allow the solution to settle in a dark place. Decant or filter the clear solution through glass wool and allow the solution to stand 20 hours before use.

(*e*) N|2 hydrochloric acid.

(*f*) 1% phenolphthalein solution in alcohol.

(*4*) *Procedure.*

(*a*) Weight accurately in a clean and dry 250 ml. Erlenmeyer flask about 1.5-2.0 gms. (about 2 ml.) of the sample taking care that no oil adheres to the neck of the flask. Note the weight of oil taken (W) gms.

(*b*) With a pipette add very carefully 25 ml. of alcoholic potash 3(*d*), counting the drops which are added after the pipette is emptied into the flask containing the oil.

(*c*) In the same manner add 25 ml. of alcoholic potash to another similar flask without oil, for a blank determination.

(*d*) Place both the flasks under reflux condensers in a water-bath and heat for about an hour rotating the flask occasionally to ensure thorough mixing, or until all the oil in the assay flask is saponified which will be indicated by the absence of any oily drop in the flask.

(*e*) Remove both the flasks from the water-bath and allow them to cool. Add 10 ml. of neutral alcohol to each of the flasks.

(*f*) Titrate both of them with N|2 hydrochloric acid using 1 ml. of phenolphthalein solution as indicator. Note the volumes in millilitres of N|2 hydrochloric acid required for the assay flask and for the blank (Y and X respectively).

(*5*) *Calculation of result.*

$$\text{Saponification value} = \frac{(X - Y) \times 28.05}{W}$$

(*6*) *Note.*

When the soap solution is dark in colour as is often the case with deep coloured oils, there is a possibility of the end-point being obscured. To avoid this a 2% alcoholic solution of Alkali Blue 6B should be used in place of phenolphthalein as indicator.

6.—Determination of iodine value.

(*1*) *Definition.*

Iodine value of an oil or fat is the percentage of iodine monochloride which is absorbed by the oil or fat expressed in terms of iodine.

(2) *General remarks.*

Wij's method for determination of iodine value is now almost universally employed and the same has been adopted. However, to ensure accuracy in results, careful attention should be paid to the following points :—

(*a*) The vessels to be used must be scrupulously clean and dry.

(*b*) The reagents used should be of the highest purity.

(*c*) The iodine solution must be carefully measured (counting drops from pipette after the liquid has run out, and using the same pipette and running in the same number of drops in blank determination).

(*d*) The iodine solution should be measured out at the same temperature.

(e) The titration should be carried out in such a way that the end-point is correct within half a drop of the thiosulphate solution.

(3) *Outline.*

A known quantity of the sample is dissolved in a suitable solvent, and excess of iodine monochloride solution added. After allowing for the absorption of iodine monochloride, the excess is estimated by the use of potassium iodide and sodium thiosulphate, with starch as indicator. The difference between the original iodine content of the solution and its final iodine content represents in terms of iodine the iodine monochloride absorbed by the sample.

(4) *Apparatus and chemicals required.*

(*a*) Iodine value flasks, 500 ml. capacity.

(*b*) Potassium iodide, A.R.

(*c*) Sodium thiosulphate, A.R.

(*d*) Starch soluble (Indicator quality).

(*e*) Glacial acetic acid, A.R.

(*f*) Iodine, A.R.

(*g*) Bleaching powder.

(*h*) Sulphuric acid.

(*i*) Potassium dichromate, A.R.

(*j*) Carbon tetrachloride, C.P.—Redistilled and dried over fused calcium chloride.

(*k*) Hydrochloric acid A.R.

(5) *Preparation of reagents.*

(*a*) *Potassium iodide solution.*—Dissolve 45 gms. of pure potassium iodide in 300 ml. of water and keep the solution in a blue or dark amber coloured bottle.

(*b*) *Starch paste.*—Take 1 gm. of powdered starch and make a thin paste with a little water, and pour the mixture into 100 ml. of boiling water. Heat for a few minutes on water-bath till quite clear. The solution should be made up fresh each day.

(*c*) *Potassium dichromate solution* N|10.—Dissolve 4.9033 gms (accurately weighed) of pure potassium dichromate in distilled water and dilute to one litre.

(*d*) *Sodium thiosulphate solution* N|10.—Boil about 15 ml. of distilled water in a flask for 30 minutes to render it sterile. After boiling, invert a small beaker over the neck of the flask while the water is cooling. Dissolve 24.82 gms. of sodium thiosulphate in the boiled water cooled to room temperature and dilute to 1 litre with distilled water—boiled and cooled.

Standardise the solution as follows :—

Place in a glass stoppered flask, 20 ml. of N|10 potassium dichromate solution and 10 ml. of 15% potassium iodide solution. Add 5 ml. of hydrochloric acid. Dilute with 100 ml. of freshly boiled and cooled distilled water and allow the N|10 sodium thiosulphate solution from burette to flow into the flask, until yellow colour of the liquid has almost disappeared. Add 1 ml. of starch solution and titrate with constant shaking until the blue colour just disappears. Calculate the strength of the sodium thiosulphate solution.

(*e*) *Wij's solution.*—Dissolve 12.7 gms. of iodine in 1 litre of glacial acetic acid* warming gently on water-bath (carefully avoiding contact of the acetic acid with steam or vapour) to effect solution. Cool the solution so obtained and titrate 10 ml. of it with standard N|10 sodium thiosulphate solution in the presence of an excess of potassium iodide and water. Place aside about 100 ml. of the iodine solution in another vessel.

Pass dry †chlorine into the iodine solution until the dark brown colour of the solution changes to clear dark orange tint. The iodine solution placed aside is now added until the colour of the solution becomes finally brown. Titrate this solution in the same way with sodium thiosulphate solution. The halogen content as determined in this second titration should be approximately, but not more than, doubled.

Heat the Wij's solution to about 100°C on a water-bath for 20 minutes and allow to cool. During the preparation of the solution access of water vapour should be prevented. Wij's solution should be kept in a glass stoppered, blue or amber coloured bottle and stored in a cool place.

Do not use Wij's solution that is more than 30 days' old.

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*When 10 ml. of concentrated sulphuric acid (1.84) containing 0.05 ml. of saturated potassium dichromate solution are added to 10 ml. of the acetic acid, the mixture should not show immediate formation of green colour.

†The bleaching powder should be mixed with twice its weight of plaster of paris and water, the paste allowed to set, and broken up into lumps. Concentrated hydrochloric acid is allowed to drip on to the bleaching powder lumps with about five times their weight of water. The chlorine gas is washed with a little water and then dried by bubbling through sulphuric acid in a wash-bottle.

(6) *Procedure.*

(*a*) Weigh accurately about 0.2 gm. of the sample in a clean, dry, well fitting glass stoppered iodine value flask of about 500 ml. capacity.

(*b*) Take another similar iodine value flask for blank determination.

(c) Add 10 ml. of carbon tetrachloride to each of the flasks shaking the latter so as to dissolve the sample in carbon tetrachloride.

(*d*) Add 25 ml. of Wij's solution to both the flasks, running out the same number of drops from the pipette after emptying.

(*e*) Insert the stoppers moistened with 15% solution of potassium iodide and gently mix the contents of the flasks with a rotary motion.

(*f*) Allow the flasks to stand for two hours in a dark place *i.e.* inside a cup-board.

(*g*) Take out the flasks and titrate each of them quickly as under :—

(*i*) Wash down very carefully the stopper and sides of the flasks, first with 10 ml. of 15% potassium iodide solution and then with 100 ml. of distilled water.

(*ii*) Titrate with standard N|10 thiosulphate solution using starch as indicator.

(7) *Calculation of result.*

If X = Volume in ml. of N|10 sodium thiosulphate solution required for blank,

Y = Volume in ml. of N|10 sodium thiosulphate solution required when the sample is used and

W = Weight in gms. of the sample taken : then

$$\text{Iodine value} = \frac{(X - Y) \times 0.01269 \times 100}{W}$$

(8) *Note.*

(*a*) Oil should preferably be weighed by means of small weighing bottles and glass tubes.

(*b*) If the oil sample is not quite clear, filter through a thick dry filter paper before weighing it.

(*c*) If the character of the oil is not known, a preliminary test is advisable as it is essential to have at least double the quantity of iodine monochloride solution than is actually required for the absorption. The weight of the oil to be taken should be accordingly adjusted.

7.—Determination of unsaponifiable matter.

(*1*) *Definition.*

The unsaponifiable matter in an oil or fat consists of those substances which are not water soluble after saponification. It is expressed as a percentage of the oil or fat.

(*2*) *Outline.*

A weighed quantity of oil is saponified with alcoholic potash and the alcohol evaporated off. The soap paste which is thus formed is dissolved in water and the unsaponifiable matter extracted in ether by means of a separating funnel. The ether is evaporated off and the unsaponifiable matter determined.

(*3*) *Apparatus and chemicals required.*

(*a*) Erlenmeyer flask, 250 ml. capacity.

(*b*) Two separating funnels preferably pear shaped, 500 ml. capacity and 250 ml. capacity.

(*c*) Reflux condenser.

(*d*) Petroleum ether (b.p. below 60°C).

(The petroleum ether, after distilling 150 ml. over 5 gms. of a refined fat or oil and drying at 100°C should leave a residue of less than 5 mgms.).

(*e*) Approx. (but not weaker than) N/2 alcoholic caustic potash.

(*f*) 95% alcohol.

(*4*) *Procedure.*

(*a*) Weigh accurately 4.0 to 5.0 gms. (W1) of the sample in a 250 ml. flask.

(*b*) Add 50 ml. of N/2 potassium hydroxide in 95% alcohol and heat for about one hour on a water-bath under a reflux condenser, occasionally rotating the flask to ensure complete saponification.

(*c*) Add 50 ml. of distilled water through the condenser and swirl to mix the contents.

(*d*) After cooling, pour the contents in a 500 ml. capacity separating funnel.

(*e*) Rinse the flask several times with petroleum ether, 50 ml. in all and add the rinsings to the solution.

(*f*) Shake vigorously for one minute so as to ensure intimate contact between petroleum ether and the soap solution. After resting and allowing the two layers to separate completely, run out the soap solution to a second separating funnel, and extract again with a further 50 ml. of petroleum ether. Separate the layers again and extract once again with 50 ml. of petroleum ether.

(*g*) Collect the three portions of petroleum ether as obtained above in a 250 ml. capacity separating funnel and wash it three times in succession with 50 ml. each of 50% alcohol.

(*h*) Transfer the petroleum ether solution to a weighed flask.

(*i*) Rinse the separating funnel with about 10 ml. of petroleum ether and transfer to the weighed flask.

(*j*) Remove the solvent by distillation.

(*k*) Dry the residual matter in an air oven at a temperature of 100-105°C to a constant weight (W2).

(*5*) *Calculation of result.*

$$\text{Unsaponifiable matter } \% = \frac{W2 \times 100}{W1}$$

(*6*) *Note.*

(*a*) If emulsion is formed during extraction with petroleum ether as happens in some cases, it is broken by adding small quantities of alcohol or concentrated potassium hydroxide or sodium chloride solution.

(*b*) Washings of ether should be continued till they are free from alkali, as shown by testing with phenolphthalein solution.

(*c*) As a check, the residue is dissolved in 10 ml. of accurately neutralised 95% alcohol and titrated with N|10 caustic soda solution using phenolphthalein as indicator. If the titration so obtained exceeds 0.1 ml. the test shall be rejected and the determination repeated from the beginning.

8.—METHODS FOR DETECTING THE PRESENCE OF DIFFERENT OILS.

(*A*) *Detection of mineral oil.*

(*1*) *General remarks.*

Mineral oils or rosin when present in vegetable oils increase the quantity of unsaponifiable matter over that normally present.

(*2*) *Qualitative tests.*

Before any quantitative determinations the following qualitative tests may be performed :—

(*a*) *Test with ultra-violet light.*

A drop of oil when seen in ultra-violet light produces a blue fluorescence if mineral oil is present in it.

(*b*) *Modified Holde rapid test.*

Apparatus and chemicals required.

(*i*) Boiling tubes.

(*ii*) Alcoholic caustic soda solution prepared by mixing 1 ml. of 50% caustic soda solution in 30 ml. of absolute alcohol.

Procedure.

(*i*) Take 15 ml. of alcoholic caustic soda solution in the boiling tube and add 0.5 ml. of the oil.

(*ii*) Boil in water-bath till the solution becomes clear indicated by the absence of any oily drops on the sides of the tube.

(*iii*) Take out the tube from the water-bath and carefully add along the sides of the tube 15 ml. of hot distilled water.

Presence of mineral oil is indicated by turbidity in solution, the depth of turbidity depending upon the percentage of mineral oil present

(*3*) *Quantitative test.*

Determine the total unsaponifiable matter of the sample as described under method 7.

Calculation of result.

Mineral oil percentage = (Total unsaponifiable matter percentage obtained)—(The average natural unsaponifiable matter percentage of the particular oil tested).

(*B*) *Detection of cotton seed oil.*

Halphen colour test.

(*1*) *Apparatus and chemicals required.*

(*a*) Hard glass test tube.

(*b*) Carbon disulphide.

(*c*) Flowers of sulphur.

(*d*) Amyl alcohol.

(*2*) *Procedure.*

(*a*) Make 1% solution of sulphur in carbon disulphide.

(*b*) Take about 2.5 ml. of the oil in a hard glass test tube and dissolve in an equal volume of amyl alcohol.

(*c*) Add 2.5 ml. of the above sulphur solution.

(*d*) Fit a cork into the mouth of the test tube and secure the cork by binding a piece of linen over it with twine.

(*e*) Heat the tube in a water-bath for thirty minutes.

In presence of cotton seed oil a deep red colour (sometimes orange red) is produced.

(*C*) *Detection of sesame oil.*

Baudouin test.

(*1*) *Apparatus and chemicals required.*

(*a*) Glass stoppered test tube.

(*b*) Freshly distilled furfurol.

(*c*) Hydrochloric acid (sp. gr. 1.19), A.R.

(*d*) Absolute alcohol.

(*2*) *Procedure.*

(*a*) Prepare a 2% solution of freshly distilled furfurol in alcohol.

(*b*) Take 5 ml. of the oil in a glass stoppered test tube and add equal volume of concentrated hydrochloric acid (sp. gr. 1.19).

(*c*) Add 8 drops of furfurol solution prepared above.

(*d*) Stopper and shake the tube vigorously for half a minute and allow to settle.

In the presence of sesame oil, the acid layer becomes red in colour. The depth of the colour which varies from light pink to deep red depends upon the quantity of sesame oil present.

(*D*) *Detection of groundnut oil.*

Bellier test.

(*1*) *Apparatus and chemicals required.*

(*a*) Round bottomed flask, 250 ml. capacity.

(*b*) Platinum wire.

(*c*) Potassium hydroxide, C.P.

(*d*) 90% alcohol.

(*e*) Acetic acid glacial, C.P.

(*f*) Hydrochloric acid, C.P.

(*2*) *Procedure.*

(*a*) Prepare alcoholic potash solution by dissolving 80 gms. of potassium hydroxide in 80 ml. of water and making it upto 1 litre with 90% (by vol.) alcohol.

(*b*) To 5 gms. of the oil in a 250 ml. flask add 25 ml. of alcoholic potash solution prepared as above.

(*c*) Boil the mixture for 5 minutes under a reflux condenser.

(*d*) To the hot soap solution, add 7.5 ml. of dilute acetic acid (1 vol. of glacial acetic acid in 2 vols. of water) and 100 ml. of acid alcohol (70% by vol. of alcohol containing 1% by vol. of hydrochloric acid).

(*e*) Cool to 12-14°C and keep there for one hour.

In the presence of groundnut oil, a precipitate is formed. No precipitate indicates the absence of groundnut oil.

(*f*) If there is a precipitate, filter and wash it with acid alcohol at 17-19°C breaking the precipitate occasionally by means of a platinum wire bent into a loop, till the washings give no turbidity or only a faint turbidity with water.

(*g*) Dissolve the precipitate in 25-70 ml. of 90% alcohol (depending upon the quantity of the precipitate) if necessary by warming it.

(*h*) Cool the solution and allow it to stand at 15°C for 1-3 hours.

Presence of groundnut oil is confirmed by a distinct precipitate at this stage. No precipitate here indicates the absence of groundnut oil.

(*3*) *Note.*

(*a*) The exact procedure must be adhered to in order to obtain reliable results.

(*b*) Some vegetable oils *i.e.* olive oil, give turbidity at first but this is not obtained on repeating after rewarming. Others give a slight opalescence, which is not followed by a distinct precipitate as in the case of groundnut oil.

(*E*) *Detection of argemone oil.*

Ferric Chloride Test.

(*1*) *Apparatus and chemicals required.*

(*a*) Hard glass test tube.

(*b*) Hydrochloric acid, C.P.

(*c*) Ferric chloride ($FeCl_3.6H_20$), C.P.

(*2*) *Procedure.*

(a) Prepare ferric chloride sollution by dissolving 20 gms. of ferric chloride ($FeCl_3.6H_20$) in 80 ml. of distilled water to which 10 ml. of hydrochloric acid are added.

(*b*) Take 10 ml. of the oil in a clean and dry test tube.

(*c*) Add 3 ml. of pure concentrated hydrochloric acid.

(*d*) Place the tube in a boiling water-bath for about two minutes and then shake it thoroughly.

(*e*) Place the tube again in water-bath for another one minute and shake it well, again.

(*f*) Place the test tube in water-bath for further 2-3 minutes till the separation of acid and oily layers is complete.

(*g*) Pipette off the oily layer as far as possible.

(*h*) Add 1 ml. of the ferric chloride solution prepared under (*a*) to the residual acid layer in the tube, slowly, along the sides of the tube.

(*i*) Give a gentle rotary motion between the palms of the hand so as not to allow the acid and the small oily layer remaining in the tube to remix.

(*j*) Place the tube again in water-bath for another ten minutes.

In presence of argemone oil, an orange red precipitate appears in the acid layer or at the interface.

(*3*) *Note.*

The precipitate obtained contains characteristic needle shaped crystals which can be observed under a microscope as a confirmatory measure.

(F) Detection of linseed oil.

Hexabromide value.

(1) Apparatus and chemicals required.

(*a*) Polenske flask, 300 ml. capacity.
(*b*) Erlenmeyer flask, glass stoppered, 250 ml. capacity.
(*c*) Separating funnel, 500 ml. capacity.
(*d*) Sintered glass filter, medium grade, 30 ml. capacity.
(*e*) Filter pump, university pattern.
(*f*) Filter flask, 1000 ml. capacity.
(*g*) Sodium hydroxide, C.P.
(*h*) Glycerine pure.
(*i*) Sulphuric acid, C.P.
(*j*) Sulphuric ether, B.P.
(*k*) Sodium sulphate anhydrous, C.P.
(*l*) Acetic acid glacial, C.P.
(*m*) Bromine pure.

(2) Procedure.

(*a*) Weigh accurately about five grams of the oil into a Polenske flask of 300 ml. capacity. Note weight (W_1) gms.

(*b*) Add 2 ml. of 50% caustic soda solution and 20 ml. of pure glycerine. Heat cautiously over a naked flame (rotating the flask by movement of the wrist) until the liquid has become clear.

(*c*) Set aside to cool, but while still hot (not over 100°C) add 50 ml. of distilled water, and rotate the flask until the soap formed is dissolved.

(*d*) Add a drop of methyl orange and gradually add approximately 5 N. sulphuric acid solution (rotating the flask) until a deep pink colour is obtained. Place the flask on water-bath until the fatty acids formed are melted.

(*e*) Cool under tap and add 30 ml. of sulphuric ether (free from alcohol). Shake until the fatty acids are dissolved and transfer to a separating funnel. Allow the mixture to separate. Drain out the lower portion into the Polenske flask and the ether solution into a glass-stoppered Erlenmeyer flask of 250 ml. capacity. Wash the aqueous portion in the Polenske flask further twice with 20 ml. of ether, collecting the etherial washings each time into the Erlenmeyer flask.

(*f*) Finally, wash the separating funnel with 10 ml. of ether, adding this washing also to the Erlenmeyer flask.

(*g*) Add about 4 gms. of anhydrous sodium sulphate to the ether extract in the Erlenmeyer flask. Allow it to stand for a couple of hours and filter the dried extract through a dry filter paper by decantation and washing the sodium sulphate residue in the flask with ether. Collect the filtrate in another 250 ml. glass stoppered Erlenmeyer flask.

(*h*) Add 10 ml. of glacial acetic acid to the flask and keep it in a cold bath at 0°C for ½ an hour.

(*i*) Keeping the flask in the cold bath add bromine slowly, drop by drop and at intervals, rotating the flask until permanent red brown colouration is obtained.

(*j*) Stopper the flask and allow to stand in the cold bath for at least four hours.

(*k*) Filter under vacuum through a tared sintered glass filter (medium grade capacity 30 ml.). The filter is kept cool during filtration by fitting it up in an ice-bath. First, decant the brown liquid, then wash the crystals with dry chilled ether and decant, and finally transfer the crystals to the filter using dry chilled ether. Keep the Erlenmeyer flask in the cold bath until the precipitate is completely transferred. Finally, wash the precipitate in the filter with dry chilled ether.

(*l*) Dry the filter in a water oven and weigh. Note weight of precipitate (W_2) gms.

(*3*) *Calculation of result.*

$$\text{Hexabromide value} = \frac{W_2 \times 100}{W_1}$$

(*4*) *Note.*

If the experiment is carried out properly, the dried hexabromide crystals are white and crumbly.

II—Special tests.

1.—Mustard oil.

(*A*) *Determination of allyl iso-thiocyanate.*

(*1*) *General remarks.*

The determination of allyl iso-thiocyanate gives the essential oil content present in mustard oil.

(*2*) *Outline.*

The essential oil in mustard oil is distilled under steam and collected in standard silver nitrate solution. From the amount of silver nitrate entering into reaction to form silver thiocyanate, the percentage of allyl iso-thiocyanate is calculated.

(*3*) *Apparatus and chemicals required.*

(*a*) Distillation flask, 500 ml. capacity.

(*b*) Measuring flask, 200 ml. capacity.

(*c*) Glass tube, 3'-4' long.

(*d*) Glass beads.

(*e*) Nitric acid, C.P. (sp. gr. 1.153).

(*f*) N|10 silver nitrate solution.

(*g*) 10% ammonia water.

(*h*) N|10 ammonium thiocyanate solution.

(*i*) 10% ferric ammonium sulphate solution.

(*j*) 90% neutral alcohol.

(*4*) ***Procedure.***

(*a*) Weigh accurately about 5 gms. (Y) of mustard oil in a dry 500 ml. capacity distillation flask and add 25 ml. of 90% neutral alcohol, 250 ml. of distilled water, a few glass beads and distil in steam.

(*b*) Collect about 150 ml. of the distillate as rapidly as possible in a 200 ml. capacity measuring flask containing 10 ml. of 10% ammonia and 25 ml. of distilled water. At the end of the distillation add 25 ml. of N|10 silver nitrate to the measuring flask.

(*c*) Fit up a 3′-4′ long glass tube to the measuring flask and heat the contents on a water-bath for one hour in a condition of active ebullition.

(*d*) Allow the mixture to cool to room temperature and make up the volume to 200 ml. with distilled water. Shake the contents and filter.

(*e*) Acidify 100 ml. of the filtrate with 6 ml. of nitric acid and add a few drops of 10% ferric ammonium sulphate solution.

(*f*) Titrate with N|10 ammonium thiocyanate till the red colour becomes permanent. Note the volume of ammonium thiocyanate used (X) ml.

(*5*) ***Calculation of result.***

$$\text{Percentage of allyl iso-thiocyanate} = \frac{(25 - 2X) \times 0.4955}{Y}$$

(*B*) ***Detection and determination of hydrocyanic acid.***

(*1*) ***Outline.***

Purified air is drawn through heated oil and absorbed in caustic potash solution to form potassium cyanide which is then determined by titration against standard silver nitrate solution.

(*2*) ***Apparatus and chemicals required.***

(*a*) Distillation flask, 250 ml. capacity.

(*b*) Absorption tube.

(*c*) Sheet of black glazed paper.

(*d*) Accurate burettes and pipettes.

(*e*) Approximately normal caustic potash solution.

(*f*) N|10 silver nitrate solution.

(*g*) 2% ferrous sulphate solution.

(*h*) 1% ferric chloride solution.

(*3*) ***Procedure.***

(*a*) Qualitative test.

Before proceeding for quantitative determination of hydrocyanic acid in mustard oil, the following qualitative test for its detection is recommended.

(*i*) Take 50 ml. of mustard oil in a distillation flask and heat on a water-bath.

(*ii*) Purify air by passing it through caustic soda and lead acetate solutions.

(*iii*) Pass the above purified air through the distillation flask kept in water-bath for about ½ an hour.

(*iv*) Absorb the outcoming air in 5 ml. of normal caustic potash solution contained in an absorption tube.

(*v*) Pour the contents of the absorption tube into a test tube and dilute with small quantity of distilled water.

(*vi*) Add a few drops of 2% ferrous sulphate and 1 drop of 1% ferric chloride solutions to the test tube.

(*vii*) Shake and warm it gently.

(*viii*) Filter after 5 minutes and add a few drops of strong hydrochloric acid.

In presence of hydrocyanic acid, a blue or bluish green colour or precipitate is formed.

(*b*) Quantitative test.

(*i*) Weigh accurately about 100 gms. (Y) of mustard oil in a distillation flask and heat on a water-bath.

(*ii*) Purify air by passing through caustic soda and lead acetate solutions.

(*iii*) Pass the above purified air through the distillation flask kept in water-bath for about 2 hours.

(*iv*) Absorb the outcoming air in 10 ml. of normal caustic potash solution contained in an absorption tube.

(*v*) Transfer contents of the absorption tube to a 250 ml. Erlenmeyer flask.

(*vi*) Rinse the absorption tube twice with about 15 ml. of distilled water each time and transfer the rinsings to the same flask.

(*vii*) Place the flask over a sheet of black glazed paper and titrate the contents with N/10 silver nitrate solution with constant stirring till a faint permanent turbidity is obtained. Note the volume of silver nitrate solution used (X) ml.

(*4*) *Calculation of result.*

Each ml. of N/10 silver nitrate solution required for the titration indicates the presence of 0.005409 gm. of hydrocyanic acid. Hence

$$\% \text{ of HCN} = \frac{X \times 0.005409 \times 100}{Y}$$

$$= \frac{X \times 0.5409}{Y}$$

2. CASTOR OIL.

(*A*) *Determination of acetyl value.*

(*1*) *Definition.*

Acetyl value is the number of milligrammes of potassium hydroxide required to neutralize the acetic acid obtained when 1 gramme of an acetylated oil is saponified.

(*2*) *Outline.*

The oil is acetylated with excess of acetic anhydride, washed and dried. Saponification values of the acetylated and unacetylated oils are determined and the acetyl value calculated by the given formula.

(*3*) *Apparatus and chemicals required.*

(*a*) Kipp's carbon dioxide generating apparatus.

(*b*) Round-bottomed flasks, 300 ml. capacity with well fitting ground glass joint reflux condensers.

(*c*) Accurate burettes and pipettes.

(*d*) Apparatus and chemicals as described under method 5.

(*e*) Acetic anhydride b.p. (137-139°C), pure and free from acetic acid.

(*f*) Approximately N/2 alcoholic potash as described under method 5.

(*4*) *Procedure.*

(*a*) Boil a mixture of 50 ml. of sample and 50 ml. of acetic anhydride for 2 hours in the acetylation flask under reflux condenser.

(*b*) Pour the mixture into a beaker containing 500 ml. of distilled water and boil for 15 minutes. Bubble a stream of carbon dioxide through the mixture during boiling to prevent bumping.

(*c*) Discontinue boiling, cool slightly and remove the water with a siphon. Add another 500 ml. of distilled water and boil again.

(*d*) Repeat as directed in (*b*) above for a third time and remove the wash water which should then be neutral to litmus.

(e) Transfer the acetylated fat to a separating funnel and wash with two 200 ml. portions of warm (60-70°C) distilled water.

(*f*) Drain and remove as much of water as possible and then transfer the fat to a beaker and add 5 gm. of anhydrous sodium sulphate. Allow to stand for 1 hour with occasional stirring.

(*g*) Filter through a dry filter paper, in an oven at 100-105°C. Remove the filter paper and keep the sample in the oven until thoroughly dry. The acetylated sample must be clear and brilliant.

(*h*) Determine the saponification values of the acetylated and un-acetylated portions as described under method 5.

(*5*) *Calculation of result.*

If S is the saponification value before acetylation and S′ the saponification value after acetylation, then

$$\text{Acetyl value} = \frac{S' - S}{1.0 - 0.00075\ S}$$

(*B*) *Determination of critical solution temperature in alcohol.*

(*1*) *Definition.*

The lowest temperature at which a vegetable oil becomes completely soluble in certain organic solvent (ethyl alcohol in this instance) is the critical solution temperature of the oil in that solvent.

(*2*) *General remarks.*

Two solvents mostly used for determining the critical solution temperatures of various oils are acetic acid and ethyl alcohol. The critical solution temperature in various solvents is a constant depending upon the nature of the glycerides composing the oil and hence gives an idea about the character of the oil.

Castor oil is soluble in ethyl alcohol, methyl alcohol, acetic acid, etc. but insoluble in petroleum ether and hydrocarbons. Other oils, however, vary to some extent in their solubility in these solvents.

Owing to the difficulty of preparing and maintaining the strength of acetic acid, the solvent ethyl alcohol is prescribed here for the test.

(*3*) *Chemical required.*

Alcohol reagent.

Dilute absolute alcohol with distilled water to such a degree that the specific gravity of the mixture at 15.5°C is 0.8303 ± 0-0001 when compared with water at the same temperature. (Once prepared the reagent will not tend to vary in strength, though it should be checked from time to time).

(*4*) *Procedure.*

(*a*) Add about 1 gm. of the oil to a clean and dry test tube.

(*b*) Mix the oil with 4.15 times its weight of the alcohol reagent.

(*c*) Allow the test tube to stand in ice-bath and cool the contents to 0°C.

(*d*) Maintain at 0°C for 5 minutes.

(*e*) Note if there is any turbidity.

(*5*) *Note.*

(*a*) Industrial methylated spirit or any form of denatured alcohol should not be used in preparation of the reagent.

(*b*) The free fatty acids present in oil lower the turbidity point. Hence both the factors should be taken into consideration.

(*c*) Duplicate determination when required should be made with a fresh portion of reagent and oil. The same mixture of oil and reagent should not be used again for the test.

(*d*) With pure castor oil, there should be no turbidity at 0°C.

3.—VANASPATI.

Determination of melting point (capillary slip method).

(*1*) *Outline.*

Melted fat is taken in a capillary tube and allowed to solidify. The tube is attached to the bulb of a thermometer in a water-bath which is gradually heated. The temperature at which the fat is seen to rise in the capillary is taken as its melting point.

(*2*) *Apparatus required.*

(*a*) Fine capillary glass tubes 5-6 cm. long, external diameter 1.2-1.5 mm. and internal diameter 0.83-1.1 mm., open at both ends

(*b*) A standardised thermometer reading in 1|5°C.

(*c*) A beaker provided with a side tube heating arrangement.

(*3*) *Procedure.*

(*a*) Clean and dry two capillary tubes.

(*b*) Melt the fat to 50°C and mix well.

(*c*) Insert the capillary tubes into the fat so that about 1 cm. long column of the fat enters the tubes

(*d*) Keep the capillary tubes in a horizontal position at a temperature of 15-17°C till the fat is just set. (During summer the tubes may be put on a perforated metal tray which is so placed inside a small water-bath containing water at 15-17°C that the bottom of the tray just touches the water).

(*e*) Place the capillary tubes in a test tube immersed in water at 15-17°C for an hour.

(*f*) Suspend the thermometer in the centre of the beaker 2 (e) to which one of the capillary tubes is attached by a rubber band, so that the lower end of the fat column is 3 cm. below the surface of the water at 10°C contained in the beaker.

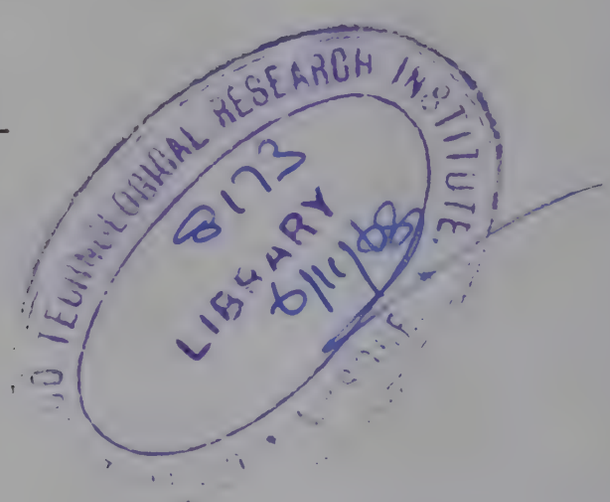
(*g*) Heat the side tube of the apparatus gently so that the temperature of water increases slowly at the rate of 2°C per minute till the temperature reaches 25°C and thereafter at the rate of ½°C per minute, continuously stirring the water in the beaker, cautiously.

(*h*) Note the temperature of water when the fat commences to rise in the tube.

(*i*) Repeat the operations from (*f*) to (*h*) above with the second capillary tube. Average of the two readings is taken as the melting point of the fat.

(*4*) ***Note.***

The melting point readings observed by two capillary tubes should not differ by more than ½°C, otherwise the test should be repeated.



Detection of Linseed Oil (Quantitative)

(1) Apparatus and chemicals required.

(a) A 25 ml. measuring cylinder or test-tube provided with a glass stopper.

(b) Bromine.

(c) Nitrobenzene

(d) Glacial acetic acid.

(2) Procedure.

(a) Dissolve 3 ml. of bromine and 12 ml. of nitrobenzene in 84 ml. of glacial acetic acid.

(b) Take about 0.5 ml of the melted oil or fat in a 25 ml. measuring cylinder or test-tube provided with a glass stopper.

(c) Add 10 ml. of the reagent made at (a) and insert the glass stopper.

(d) Shake the mixture thoroughly for about 2 minutes or until such time that a homogeneous solution is obtained and then allow it to stand for 1 or 2 hours.

If an insoluble precipitate is formed, it is indicative of the presence of Linseed Oil (or of oils containing a high percentage of Linolenic or similar highly unsaturated acids).

List of Priced Publications Issued by the Agricultural Marketing Adviser to the Government of India

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